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**COMPOSITES FOR ELECTROCHEMICAL TECHNOLOGY  
PART I. SURVEY OF ELECTRODE CORROSION  
STUDIES FOR LEAD-ACID BATTERIES**

BY I. A. ANGRES  
R. A. SUTULA

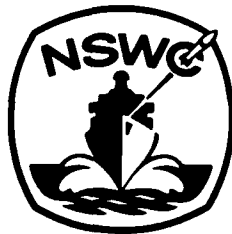
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FOREWORD

This report briefly describes corrosion methods used to screen lead electrodes which are potentially useful for submarine lead acid batteries.

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## INTRODUCTION

This report provides a brief review of corrosion literature to establish a data base on corrosion studies for lead electrodes.

The main storage battery in nuclear submarines is lead-acid, and serves the following purposes:

1. Provides a ready source of standby power capable of sustaining ship or nuclear reactor vital auxiliaries without interruption of service in the event of loss of normal power sources; and
2. In the event of a nuclear reactor scram, it sustains submerged ship operations to re-start the nuclear reactor. This includes providing propulsion, life support, ship and nuclear reactor auxiliary power.

The importance of the main storage battery in maintaining the reliability of the nuclear propulsion plant cannot be overemphasized.

The electrode grids of most lead-acid batteries are made from alloys such as lead-antimony, lead-arsenic, or lead-calcium, that possess better mechanical strength and are easier to handle than pure lead.

Present-day nuclear submarine batteries employ grids made of lead-calcium alloys. However, these grids suffer serious mechanical failure as a result of corrosion. After about 66 months of service, the positive grid of the nuclear submarine battery loses its strength and expands, causing battery failure.

The grid material of choice, from an electrochemical standpoint, is pure lead metal. It is known that pure lead has many desirable properties:

1. High hydrogen overpotential;<sup>1</sup>
2. Avoids adverse electrochemical side effect of alloying ingredients; and
3. Excellent corrosion resistance to sulfuric acid.<sup>2</sup> Pure lead, however, lacks the tensile and creep strength to serve alone as a grid material.

- 
1. Hampel, C. A., "The Encyclopedia of Electrochemistry", Reinhold, New York (1964) pg. 869.
  2. Perkins, J., Pokorny, J. L., Coyle, M. T., AD-A 032-299 "A Review of Materials and Mechanisms which affect the performance of Lead-Acid Batteries", Oct. 1976.



Exploratory development has shown that metal matrix composite grids made from pure lead reinforced with non-metallic fibers will combine the desirable electrochemical properties of pure lead with the great mechanical strength of the fibers.

Since corrosion studies are used to screen the composite materials for use as grids in lead-acid batteries, a short literature review on corrosion of lead and its electrodes including experimental details are described below.

#### CORROSION CONSIDERATIONS

Lead and its alloys are used in a variety of lead-acid battery designs and applications. Therefore, it is necessary to examine the behavior of a given alloy under the conditions of its prospective use, for example float charge.

Lander was the first to study the anodic corrosion of lead in detail. Combining a thermodynamic and kinetic approach, he related the mechanism of the positive plate reactions to the actual conditions of battery service. Lander's work showed that the grid of the  $Pb/PbO_2$  electrode is passivated at a potential corresponding to mild overcharge; which is encountered in battery-"Float" service. The original work of Lander<sup>3</sup> showed that at constant potentials in the range 0.1 to 1.0 volt, tetragonal  $PbO$  is formed in contact with lead in sulfuric acid solutions. An outer layer of  $PbSO_4$  is formed, probably by chemical reaction between  $H_2SO_4$  and the  $PbO$  layer. The corrosion rate in this voltage range increased with increasing temperature and with decreasing acid concentration. The latter effect is explained by showing a linear relation between rate and the square of the activity of water in the electrolyte. At potentials from 1.20 to about 1.50 a protective film is formed, presumably consisting of  $PbO_2$ .

Subsequently, Lander<sup>4</sup> showed that  $PbO_2$  undergoes a solid phase reaction with  $Pb$  to form tetragonal  $PbO$ . A mechanism involving the Volta effect is proposed. A contact potential between  $Pb$  and  $PbO_2$  may be an important part of the cell potential and explain self-discharge and the discharge reaction at the positive plate.

An intermediate film of  $PbO_2$  is formed by the corrosion process which does not cycle completely to  $PbSO_4$ . Because of this, it retains its compact nature and may in large measure be responsible for slowing down grid corrosion.

Grid growth appeared to be proportional to the depth of corrosion after an initial period. The initial period and the rate of growth are probably characteristic of the tensile strength or creep resistance of the metal.

The mechanisms governing rate have not been determined in all the potential ranges concerned; indeed, the whole picture is rather complicated because of the number of reactions occurring. The following reasoning can be ascertained from Lander's work:<sup>5</sup>

3. Lander, J. J., J. Electrochem. Soc., 98, 213 (1951)
4. Lander, J. J., ibid, pg. 98, 220 (1951)
5. Lander, J. J., ibid, pg. 103, 1 (1956)

1. Formation of lead dioxides by the reaction,  $\text{Pb} + 2\text{H}_2\text{O} \rightarrow \text{PbO}_2 + 4\text{H}^+ + 2\text{e}^-$ , is the first step in the corrosion process above the reversible potential for that reaction. It is unstable both on chemical and electrochemical grounds at the lower potentials and goes to lead monoxide and lead sulfate.
2. A voltage limit above which the solid-phase reaction,  $\text{Pb} + \text{PbO}_2 = 2\text{PbO}$ , cannot go has been shown to be about +1.58v to the standard hydrogen electrode. This voltage marks the appearance of lead dioxide in the corrosion product and is characterized by a peak in the corrosion-rate vs potential curve which separates the kinetic behavior of the corrosion processes. At more noble potentials, just below the reversible positive-plate potential, corrosion rates are limited by penetration of the positive-plate-discharge-reaction into an underlying lead dioxide film. This (pretration) is controlled by diffusion of  $\text{SO}_4^{2-}$  through a porous film of mixed lead dioxide and lead sulfate. At slightly less noble potentials, diffusion control gives way to a relatively rapid process because the solid phase reaction begins to take place and the lead dioxide film is attacked from both sides.
3. Above the reversible  $\text{PbO}_2/\text{PbSO}_4$  potential, corrosion rates fall off very rapidly to extremely low values, because a protective lead dioxide film is built up. Rates in this potential range do not vary with potential at room temperature within experimental error, but they do increase with increasing temperature and decreasing acid concentration.

The experimental details of Lander's work are as follows: Granular lead (purity about 99.97%) was melted in a clean glass beaker, cast into a steel mold, and cold rolled to a thickness of 0.051 cm (0.02 in.) between polished steel rolls. Samples were always cut to the same dimensions giving a total area of 34 cm<sup>2</sup> and were maintained at several constant potentials in sulfuric acid solutions in the range covering about one volt below the reversible potentials for the  $\text{PbO}_2 - \text{PbSO}_4$  couple. The sulfuric acid was used in concentration of 10, 30, and 40 percent by weight, and two constant temperatures: 30° and 50°C were employed. The rates of attack were followed either by stripping the film in ammonium acetate solution and determining weight loss directly or by recording the currents flowing through the samples. By checking measured weight losses against those calculated from Faraday's law, it was established that the law, calculated for the reaction:  $\text{Pb} \rightarrow \text{Pb}^{2+} + 2\text{e}^-$  applies at potentials below those for  $\text{PbO}_2$  formation. X-ray analyses of the film substance were also obtained.

The circuit diagram of the apparatus used for maintaining constant potential is shown in Figure 1. A resistor  $R_1$ , consisting of several feet of copper wire, was connected in series with the variable resistors  $R_3$  and  $R_4$ , so that currents producing potential drops up to 1.1 volts across  $R_1$  could be made to flow. B, the sample to be anodized, was contained in a beaker of sulfuric acid electrolyte, and could be connected to any position on  $R_1$  through  $R_2$ , a two-ohm resistor, from which leads ran to a G.E. potentiometer recorder. A and A' were sections of positive plate from a lead-acid cell, having a capacity such that currents of the magnitude drawn by the samples produced no polarization. They were connected by switches  $S_1$  and  $S_2$  to either end of the slide wire so that a given potential, anodic or cathodic to that of the  $\text{PbO}_2$  plate, could be applied to the sample. C was a mercury-mercurous sulfate reference electrode, the tip of which was kept within 1 mm of the sample. The contribution to the potential by the ohmic resistance

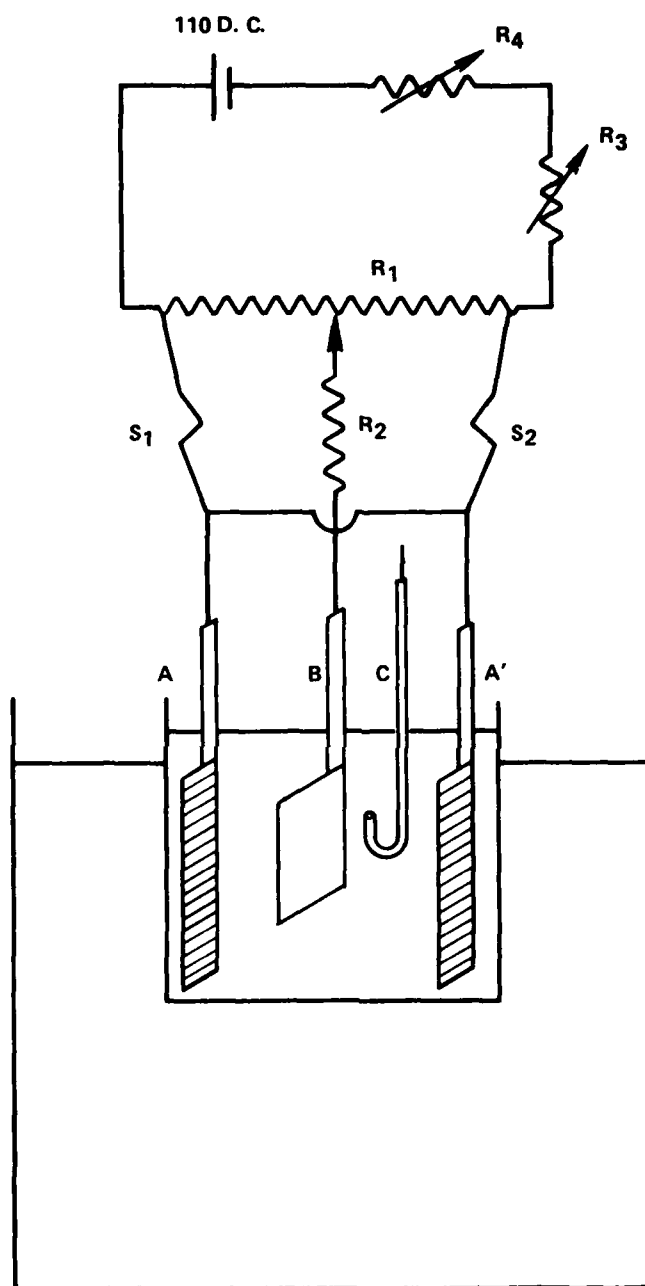
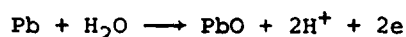


FIGURE 1 DIAGRAM OF APPARATUS

between sample and reference electrode was estimated to be considerably less than 1 mv for a current of 10 ma. The reference electrode and the sample were connected to a Rubicon Null-point potentiometer. The sample potential was adjusted manually by sliding the contact from  $R_2$  along  $R_1$  and by adjusting the resistors  $R_3$  and  $R_4$ . Potentials were controlled to within  $\pm 0.01$  volt and variations within this range did not introduce significant errors. Current-time curves were plotted from the record, and weight-loss vs. time curves were then calculated using small increments of the area under the curve and Faraday's law.

All voltages are referred to the mercury-mercurous sulfate electrode in 40.8 percent sulfuric acid at 20°C, which has a potential of +0.566 to the standard hydrogen electrode. For purposes of orientation the reversible potentials of the several couples under the various experimental conditions with respect to this electrode are given in Table I. Standard potentials are also included. These values were all calculated from the data of Harned and Hamer<sup>6</sup> except for the Pb, PbO, and Pb, Pb(OH)<sub>2</sub> couples for which  $E^\circ$ 's were calculated using the free energy data from Latimer<sup>7</sup> and the other values using the Nernst equation for the reactions:



and

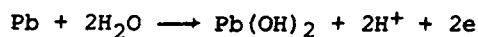


Table I. Potentials of various electrodes referred to the Hg, Hg<sub>2</sub>SO<sub>4</sub> electrode in 40.8% H<sub>2</sub>SO<sub>4</sub> at 20°C

| Electrode                            | $E^\circ$ | Concentration of H <sub>2</sub> SO <sub>4</sub> |        |        |        |        |        |
|--------------------------------------|-----------|-------------------------------------------------|--------|--------|--------|--------|--------|
|                                      |           | 10%                                             |        | 30%    |        | 40%    |        |
|                                      |           | 30°C                                            | 50°C   | 30°C   | 50°C   | 30°C   | 50°C   |
| PbO <sub>2</sub> , PbSO <sub>4</sub> | +1.685    | +1.063                                          | +1.058 | +1.135 | +1.131 | +1.181 | +1.177 |
| Hg, Hg <sub>2</sub> SO <sub>4</sub>  | +0.615    | +0.104                                          | +0.099 | +0.038 | +0.031 | +0.000 | -0.004 |
| Pb, PbO                              | +0.250    | -0.348                                          | -0.351 | -0.306 | -0.306 | -0.280 | -0.277 |
| Pb, Pb(OH) <sub>2</sub>              | +0.242    | -0.357                                          | -0.360 | -0.312 | -0.312 | -0.280 | -0.277 |
| Pb, PbSO <sub>4</sub>                | -0.355    | -0.865                                          | -0.873 | -0.933 | -0.942 | -0.970 | -0.979 |

Thomas and Haring<sup>8</sup> established that by static discharge the Pb-Sb alloy sulfates in approximately 1/9 of the time required for pure Pb and 1/10 the time required for the Pb-Ca alloy. They concluded that anodically and cathodically, the behavior of

6. Harned, H. S., and Hamer, W. J., J. Amer. Chem. Soc., **57**, 33 (1935)

7. Latimer, W. M., "Oxidation Potentials," pp 304-305, Prentice Hall Inc., NY (1938)

8. Haring, H. E., Thomas, U. B., Trans. Electrochem. Soc., **68**, 293 (1935)

the Pb-Ca alloy is similar to that of pure lead but quite distinct from that of the Pb-Sb alloy.

With respect to carbon-reinforced lead electrodes, only preliminary work has been done by Gould, and the results are encouraging.<sup>9</sup> In summary, Gould found that the best results were obtained with the HM/PVA-3000 graphite-fiber composite rods with 15 mil electroplate of lead. The rods completed 75 days on test at 71°C which would be equivalent to 2250 days (6.14 years) at room temperature. The maximum depth of corrosion was found to be three mils.

The apparatus used in these corrosion studies are shown in Figure 2. The constant potential was supplied by a custom-built multistation potentiostatic voltage regulator. A primary reference voltage was provided by a Lambda LCS-2-01 power supply with each individual station provided with a 20 turn pot for fine regulation. The sensing and current leads were separate. The voltage was checked and adjusted as required at 24-hour intervals. The corrosion studies were conducted at 71°C in 1.285 specific gravity sulfuric acid with constant potential of 1.226 v anodic to the mercury-mercurous sulfate electrode.

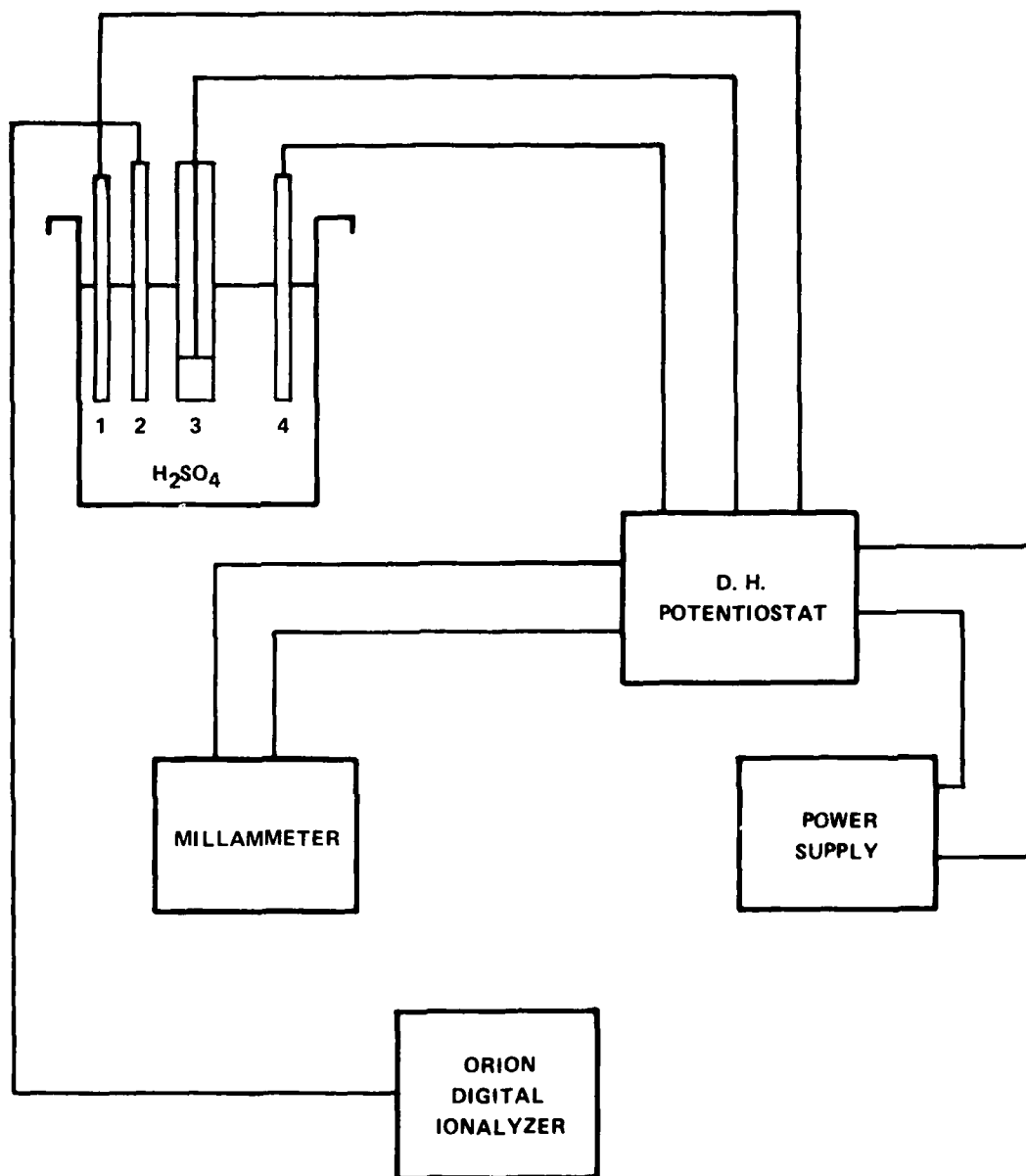
In comparing Lander's experimental set up and the experimental set up of Gould it is found that the Gould approach provides corrosion data in terms of life-stability at a much more rapid rate than Lander's approach. Lander's approach is closer to the actual corrosion a battery experiences during charge-discharge. However, it appears that a much longer time is needed to get the necessary data for final evaluation of lead composite samples.

The data obtained from the accelerated anodic corrosion studies at 71°C was assumed to follow the Arrhenius equation,

$$\frac{1}{T_1} - \frac{1}{T_2} = K \log \frac{I_2}{I_1} \quad (1)$$

where I represents the rate of any specific chemical process, and extrapolated to 25°C to obtain the service life of the composite electrode. Extrapolation of anodic corrosion data at high temperatures to room temperature must be taken with "a grain of salt." It is possible that at the high temperature the reactions and products may be substantially different than those at 25°C. Without a correlation between the 71 and 25°C temperature data, it is not possible to put any confidence in the values obtained for service life of the electrode. The accelerated anodic corrosion tests at 71°C can be used as a relative method to screen potential fiber reinforced lead composites. Since all electrodes are treated in the same way during the corrosion test, then those electrodes that last the longest would tend to give the longest service life at 25°C. Thus, it is evident that corrosion data at 25 and 35°C must be obtained to determine whether the data obtained from the accelerated anodic corrosion tests at 71°C can be modeled by equation (1) with any confidence in the service life values at 25°C.

- 
9. Mao, G. W., Fiedler, A. F., "Feasibility Study for Utilization of Fiber Reinforced Lead Composites in Naval Hardware Applications," Report No. NSWC-76-C-0215-U, 15 November 1977.



1. WORKING ELECTRODE - Pb/Ca OR LEAD COMPOSITE
2. ION SELECTIVE ELECTRODE
3. REFERENCE ELECTRODE - Hg/Hg<sub>2</sub> SO<sub>4</sub>
4. COUNTER ELECTRODE - Pb

FIGURE 2 DIAGRAM OF APPARATUS FOR INVESTIGATING THE ANODIC CORROSION OF METAL MATRIX MATERIALS

Therefore, using the apparatus of Figure 2 we recommend that accelerated anodic corrosion tests on lead, lead alloys, and fiber reinforced lead composites be performed at 25, 35 and 70°C. By performing the accelerated anodic corrosion test at three temperatures it will be possible to determine what correlation exists between the 70° and 25°C temperature data.

CONCLUSION

Accelerated anodic corrosion tests can be used to evaluate the service life of lead-acid battery electrodes provided a correlation exists between high and room temperature data. Without such a correlation the accelerated anodic corrosion tests can only be used as a relative method to screen potential fiber reinforced lead electrodes.



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